Nuclear Inst. and Methods in Physics Research, A 919 (2019) 68–72

Contents lists available at ScienceDirect

Nuclear Inst. and Methods in Physics Research, A

journal homepage: www.elsevier.com/locate/nima

Study of the fish fossil *Notelops brama* from Araripe-Basin Brazil by Neutron Tomography

Reynaldo Pugliesi [a,*], Marco A. Stanojev Pereira [a], Marcos L.G. Andrade [a], Juliana M.L. Basso [b], Cibele G. Voltani [b], Ivone C. Gonzales [b]

[a] *Brazilian Institute for Nuclear Technology IPEN-CNEN/SP Av. Prof. Lineu Prestes, 2242 – Butantã - São Paulo — SP, 05508-000, Brazil*

[b] *Geosciences Institute of University of São Paulo IGc – USP R. do Lago, 562 - Butantã, São Paulo — SP, 05508-080, Brazil*

ARTICLE INFO

Keywords:
Neutron tomography
Paleontological samples
Fish fossil specimen

ABSTRACT

In the last decade, Neutron Tomography significantly gained importance as an analyzing tool to inspect samples of paleontological interest. In general, neutrons are able to provide new and complementary information when compared to the ones provided by X-rays imaging methods. Specifically, in the present paper, the Neutron Tomography equipment of the IPEN-CNEN/SP was successfully applied to investigate the internal structure of a fish fossil, embedded in a calcareous concretion. The resulting images, which will be presented, has revealed different contrast between several regions within the specimen enabling the differencing of osteological structures of the fossil, information about its state of conservation, degree of embedding, inclusions as well as regions within the concretion which could be related to humidity or to other substances and minerals containing hydrogen naturally added to the fossil. The technique is non-destructive and non-invasive allowing a close analysis of the specimen. Furthermore, the obtained images have demonstrated the feasibility of the equipment at IPEN-CNEN/SP for investigating this kind of sample.

1. Introduction

Neutron Tomography — NT has demonstrated for being a valuable investigation tool in many fields of research such as biology, medicine, engineering, etc. Its use for archaeological and paleontological studies is quite recent and the first results were reported in the 90's [1–5]. Particularly for the interest of the present work, and in contrast to X-rays, neutrons are able to penetrate thick layers of some rocks and on the other hand provide a high sensitivity to detect small amounts of hydrogenous substances, making NT a unique tool to investigate and to characterize the internal structure of fossil samples. It is not surprising the growing interest of the scientific community to its use for such investigations [6–8].

The Brazilian Institute for Nuclear Technology (IPEN-CNEN/SP) has equipment for Neutron Tomography, which has been operational since 2011 providing images with sufficient quality, in terms of spatial resolution and sensitivity, for paleontological investigations. The results obtained in the present work have demonstrated the potential of the NT and of the present equipment to investigate fossils embedded within a rock matrix, specifically a fish fossil specimen embedded within a calcareous concretion [9–11].

2. The equipment for Neutron Tomography

The equipment for Neutron Tomography is installed at the Beam-Hole (BH) #14, of the 5 MW IEA - R1 Nuclear Research Reactor, of the IPEN - CNEN/SP. The Fig. 1 shows the equipment sketch, and some of its main components are: the rotating table where the sample to be tomographed is irradiated; the scintillator screen (Li^6F/ZNS - NE426) where the image of the internal structure of the sample is formed; the plane mirror that reflects this image to the video camera; the CCD video camera (ANDOR Ikon – M, 16 bits, 1024 × 1024 pixels, 12 x 12 μm^2 pixel size) for imaging capture; the radiation shielding. The rotating table is able to rotate the sample between 0 and 360°. The camera and the rotating table are coupled to a computer making the system automated, in such a way that after the first image being captured, the sample is rotated and a new image is captured. At present, 400 images are necessary for a tomography, the angle between images is 0.9° and the time per image capture is 1 s. Two softwares are used, the Octopus V8.7 [12,13] for image reconstruction in the viewing planes XY, XZ, YZ, and the VGStudioMAX V2.2 for 3D visualization [14]. The neutron flux at the irradiation position (measured by the Au-foil method) is 8 x

---

[*] Corresponding author.
E-mail address: pugliesi@ipen.br (R. Pugliesi).

https://doi.org/10.1016/j.nima.2018.12.001
Received 5 September 2018; Received in revised form 29 November 2018; Accepted 1 December 2018

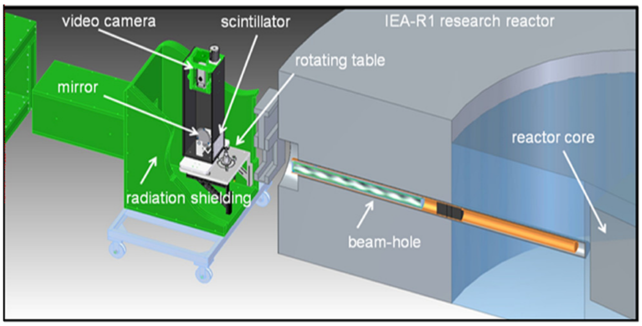

Fig. 1. Sketch of the equipment for Neutron Tomography.

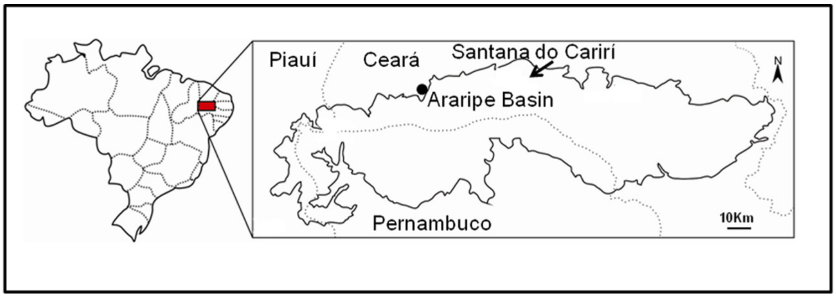

Fig. 2. Geographic location of the Araripe Basin.

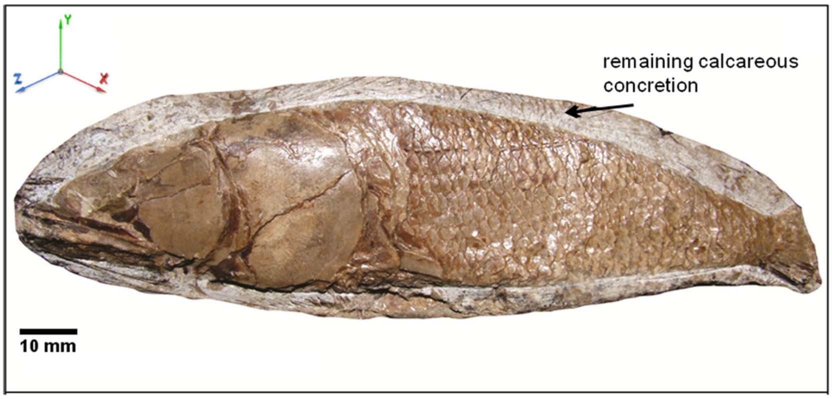

Fig. 3. Photography of the *Notelops brama*.

10^6 n cm^{-2} s^{-1}, the flat field of the beam is 16 cm in diameter, the best achievable spatial resolution is 205 ± 25 μm, and the field of view is 18 x 18 cm^2. The references [9–11,15–17] describe in detail, the equipment and the procedures for tomography.

3. The specimen

The fish fossil specimen investigated in the present study was the *Notelops brama* provided by the Geosciences Institute of University of São Paulo (IGc – USP). It was collected in the Santana Formation (Member ROMUALDO) located close to "Santana do Cariri" county at the Araripe Basin, with an area of about 8.000 to 9.000 km^2, bordering the states of Ceará, Pernambuco and Piauí in Northeastern of Brazil (Fig. 2). Among the Geological Formations in the Araripe Basin, this is the most fossiliferous with eocretacic age between 100.5 and 66 M.a [18]. From the 32 fish fossil specimens usually found in this Formation, 28 are embedded in calcareous concretions. Frequently in these specimens, the extremities of the fossils are not visible being necessary the employment of invasive techniques to remove part of the calcareous concretion for its identification [19]. Among these techniques is the partial immersion of the specimen in an acid solution, and the procedure for removing is very slow taking typically about 4 months to be concluded. In spite of being efficient, displacements of some osteological structures of skeleton are often expected, changing their original position or even damaging them [19]. The second technique, the one employed in the specimen of the present paper, the concretion was removed by scrapping, using a burin, resulting in the sample shown in Fig. 3 with full length of 147 mm and maximal width of 44 mm.

4. Results and discussions

The neutron tomography file consists of 400 slices corresponding to the plane of view XY, 150 to the plane XZ and 102 to the plane YZ, and each slice refers to a thickness of 0.28 mm of the specimen(for reference see Fig. 3). The Fig. 4a, b and c show three selected slices. The first refers to the plane XY at 37 mm depth within the specimen and the second to the plane XZ at 28 mm depth, in which it is possible to visualize the border (red dashed lines) limiting the concretion (left) and details of osteological structure of the fossil (right), as well as concretion inclusions to the fossil, here seen as light gray color blurred

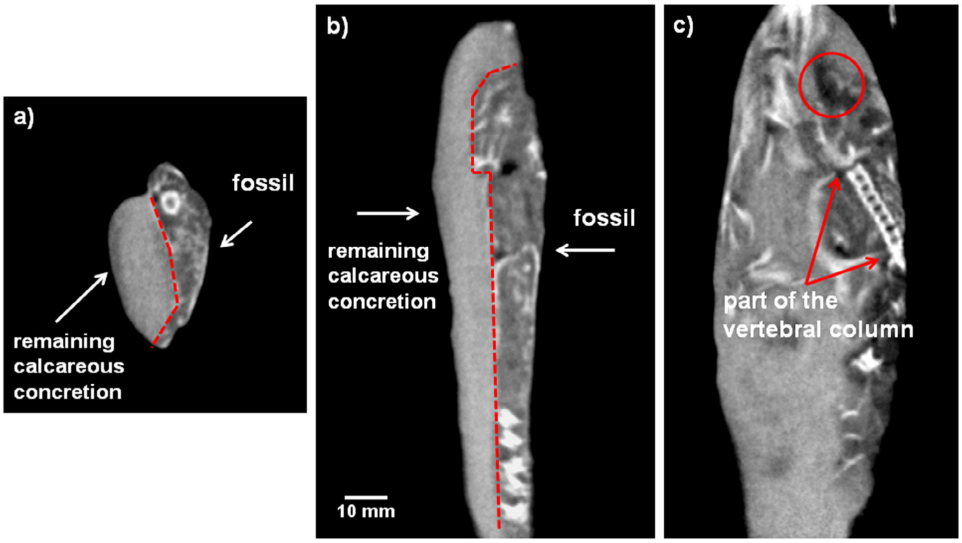

Fig. 4. Selected tomography slices for the viewing planes: (a) XY, (b) XZ, (c) YZ respectively.

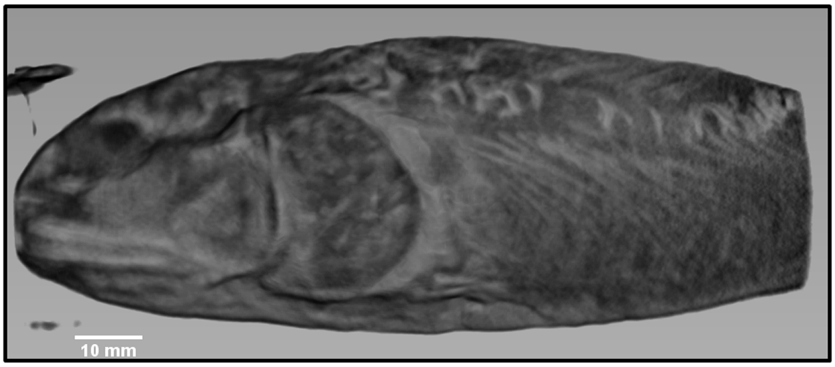

Fig. 5. 3D image obtained by Neutron Tomography.

areas. Fig. 4c shows the third selected slice, corresponding to the plane YZ, at 13 mm depth in the specimen, in which part of the osteological structure, particularly the vertebral column of the fossil as well as its Orbit (red circle) are clearly visible.

It is worthwhile to mention that, as a first qualitative analysis of the tomography slices, areas of different neutron attenuation can be clearly identified, in such way, that the brighter the area, the higher is the attenuation. These areas could be related to humidity or other substances and minerals containing hydrogen naturally added to the fossil [20]. On the other hand, void structures, here identified as darkest areas in the fossil, as in Fig. 4b, were also found. Both the voids as the bright areas were seen in tomographic slices at different depths of the specimen and their sizes can eventually be determined quantitatively. In general, for paleontology studies, such information can be a good hint to evaluate the history of the specimen [20,21].

Fig. 5 is a 3D general image of the studied specimen, obtained from the tomography file.

Using image processing tools available in the software mentioned in (2), the remaining calcareous concretion (see Fig. 3) was partially removed and some osteological structures of the fossil, shown in Fig. 6, were identified: *cleithrum* (Cl), hiomandibulare (Hm), interoperculum (Iop), *metapterygoid* (Mtp), preopercle (Pop), supracleithrum (Scl), ribs (r), dentary (D), Maxilla (Mx) and some vertebras (v). In addition, the ocular cavity (Orb) is also clearly visible [20].

In Fig. 7 the calcareous concretion was entirely removed and the entire vertebral column could be visualized. In Fig. 8 the vertebral column was zoomed, allowing clearly verifying the shape and the size of a single vertebra. From these figures, it was possible to find out the poor state of conservation of the fossil, evidenced by articulated and misaligned vertebral centers, as well as the absence of some skull bones [20].

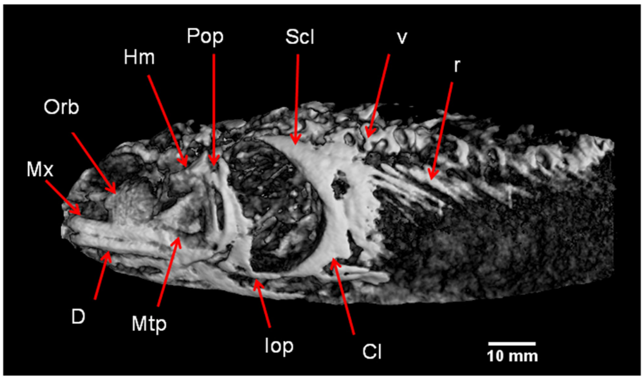

Fig. 6. 3D processed image, enhancing some osteological structures.

Searching for fragments of the missing bones that would be embedded within the remaining calcareous concretion, the 3D general image of the specimen, specifically the part referring to the concretion, was visually inspected. For such purpose this image was split in about 350 individual slices along the plan of view XY (see reference in Fig. 3), each one representing 0.28 mm of the specimen. In order to

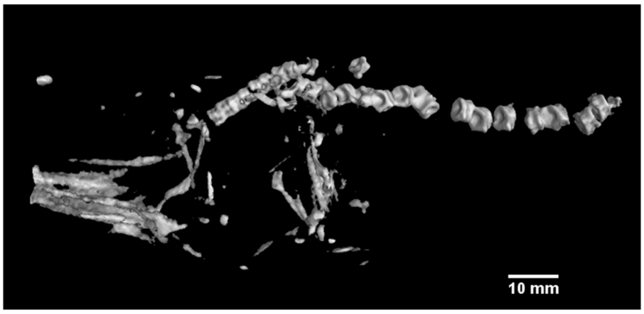

Fig. 7. 3D image enhancing the remaining osteological structure.

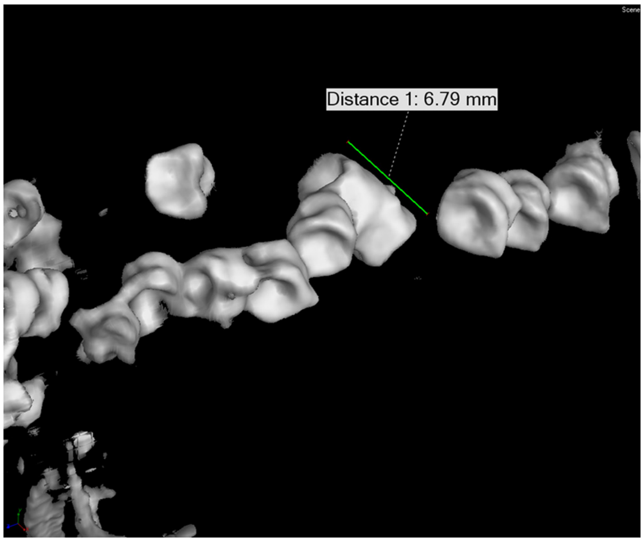

Fig. 8. Zoom of some vertebras.

facilitate the search, these slices were colored in such way the embedded fragments would appear like individual "red grains", in a "yellow-green" concretion. No fragments were found, however as demonstrated in Fig. 9, which is a 3D image of the specimen, split in half, only dispersed "red spots" in the concretion could be seen.

Finally, in performing the tomography is important to consider that the chemical elements of the specimen will be irradiated in an intense neutron beam and some of them will be activated. By scanning the gamma radiation dose rate at the surface of the specimen, the induced activation became negligible 24 h after completing the tomography process.

5. Conclusions

The tomographies, as well as the 3D images obtained in the present work, corroborate the capability of the NT technique to investigate and characterize the internal structure of fossil specimens, addressing it in a large number of paleontology problems enabling:

– study of the fossil without displacing any internal structures;

– quickness, since a tomography is obtained in 6 or 7 min;

– the study of large samples, max. 100 cm^2 with a thickness between 5 and 10 cm;

– in spite the time interval, for which the induced radioactivity by neutrons in the specimen becomes negligible, is about 24 h from the end of the tomography, It is very important to emphasize that this result is valid only for this specific specimen. For other specimens, their basic compositions and impurities, can lead to different, radiation levels and time intervals.

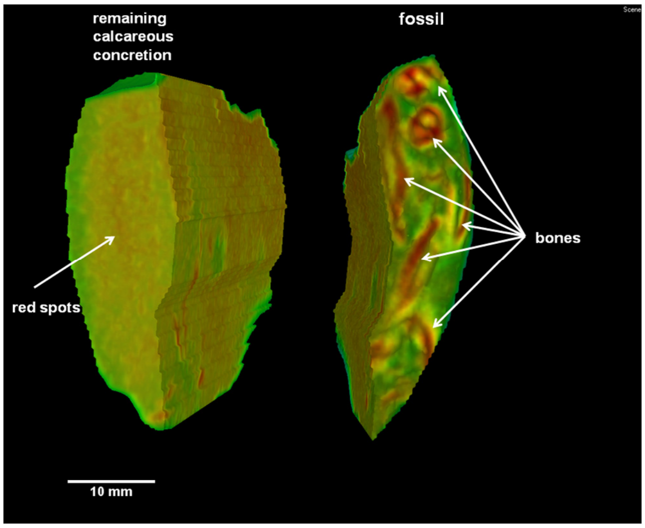

Fig. 9. 3D split image of the specimen: calcareous concretion (left) and the fossil (right) . (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Finally, the NT also enables a non-invasive previous investigation about the state of conservation and the degree of embedding of the fossil, aiming to take the decision about the necessity as well as to determine the most adequate method for removing the calcareous concretion, to expose the fossil.

Acknowledgments

The authors are indebted to the National Council of Technological and Scientific Development — CNPq (Brazil) for the post doctoral fellowship and partial financial support to this project through the Fellowship 114862/2015-0.

References

[1] J. Karch, J. Dudák, J. Žemlicka, D. Vavrík, I. Kumpová, J. Kvacek, Z. Hermanová, J. Šoltés, L. Viererbl, M. Morgano, A. Kaestner, P. Trtíkf, X-ray micro-CT and neutron CT as complementary imaging tools for non-destructive 3D imaging of rare silicified fossil plants, in: 19th Int. Workshop on Rad. Imaging Detectors 2–6 July, AGH University of Science and Technology, Krakow, Poland, 2017.

[2] G.P. Odin, V. Rouchon, F. Ott, N. Malikova, P. Levitz, L.J. and, Neutron imaging investigation of fossil woods: non-destructive characterization of microstructure and detection of in situ changes as occurring in museum cabinets, Foss. Rec. 20 (2017) 95–103.

[3] C. Mays, J.J. Bevit, J.D. Stilwell, Pushing the limits of neutron tomography in palaeontology: Three dimensional modelling of in situ resin within fossil plants, Palaeontol. Electron. 20.3.57A (2017) 1–12.

[4] F.C. De Beer, Paleontology: Fossilized ancestors awaken by neutron radiography, in: N. Kardjilov, G. Festa (Eds.), Neutron Methods for Archaeology and Cultural Heritage. Neutron Scattering Applications and Techniques, Springer, Cham, 2017, pp. 141–171.

[5] E. Deschler-Erb, E.H. Lehmann, L. Pernet, P. Vontobel, S. Hartmann, The complementary use of neutrons and X-rays for the non-destructive investigation of archaeological objects from Swiss collections, Archaeometry 46 (2004) 647–661.

[6] Kaloyan. A.A., E.S. Kovalenko, A.V. Pakhnevich, K.M. Podurets, The contrast scale of minerals for neutron tomography of paleontologic and geologic objects, Russ. Geol. Geophys. 58 (2017) 1435–1440.

[7] A.A. Kaloyan, E.S. Kovalenko, A.V. Pakhnevich, K.M. Podurets, S.V. S. V. Rozhnov, V.A. Somenkov, Synchrotron and neutron tomography for the investigation of paleontological objects, J. Surf. Invest. 8 (6) (2014) 1093–1099.

[8] D. Schwarz, P. Vontobel, E. Lehmann, C.A. Meyer, G. Bongartz, Neutron tomography of internal structures of vertebrate remains: A comparison with X-ray computed tomography, Palaeontol. Electron. 8 (2) (2004) 1–11.

[9] M.A. Stanojev Pereira, R. Schoueri, C. Domienikan, F. Toledo, M.L.G. Andrade, R. Pugliesi, The neutron tomography facility of IPEN-CNEN/SP and its potential to investigate ceramic objects from the Brazilian cultural heritage, Appl. Radiat. Isot. 75 (2013) 6–10.

[10] R. Schoueri, C. Domienikan, F. Toledo, M.L.G. Andrade, M.A. Stanojev Pereira, R. Pugliesi, The new facility for neutron tomography of IPEN-CNEN/SP and its potential to investigate hydrogenous substances, Appl. Radiat. Isot. 84 (2014) 22–26.

[11] R. Schoueri, Design and Installation of a Neutron Tomography Facility at IPEN-CNEN/SP (MSc Thesis), University of São Paulo, Brazil, 2017.

[12] M.A. Stanojev Pereira, Neutron imaging: 30 years experiencing at IPEN-CNEN-SP, Sagitarius Ed. São Paulo, ISBN: 978-85-923404-1-4, 2017.

[13] Octopus, "Octopus 8.6." 2011. <https://www.octopus.be/nl> access September, 2018.

[14] Volume Graphics, "VG Studio Max 2.2." Heidelberg; Germany. <https://www.volumegraphics.com/> access September, 2018.

[15] R. Pugliesi, M.A. Stanojev Pereira, R. Schoueri, Method to evaluate the L/D ratio of neutron imaging beams, Braz. J. Radiat. Sci. 5 (2017) 1–7.

[16] R. Pugliesi, M.L.G. Andrade, M.S. Dias, P.T.D. Siqueira, M.A. Stanojev Pereira, Study of pixel damages in CCD cameras irradiated at the neutron tomography facility of IPEN-CNEN/SP, Nucl. Instrum. Methods Phys. Res. Sect. A Accel. Spectrom. Detect. Assoc. Equip. 804 (2015) 59–63.

[17] M.A. Stanojev Pereira, R. Pugliesi, Penetration of the consolidant Paraloid® B-72 in Macuxi indigenous ceramic vessels investigated by neutron tomography, Nucl. Instrum. Methods Phys. Res. Sect. A Accel. Spectrom. Detect. Assoc. Equip. 889 (2018) 118–121.

[18] M.L. Assine, Araripe Basin, Bull. Geosci. Petrobrás 15 (2007) 371–389.

[19] H.A. Toombs, A.E. Rixon, The use of acids in the preparation of vertebrate fossils, Curator, 1959, pp. 304-312.

[20] C.G. Voltani, Commented Osteological Redescription and Paleoecological Aspects of the Genus Notelops and Rhacolepis (Pachyrhizodontoidei) (Ph.D. thesis), University of São Paulo, Brazil, 2016.

[21] Nikolay Kardjilov, Fabrizio Fiori, Giuseppe Giunta, André Hilger, Franco Rustichelli, Markus Strobl, Neutron tomography for archaeological investigations, J. Neutron Res. 14 (1) (2006) 29–36.